Characterization of dual matrix structured ductile iron

A Thesis submitted in partial fulfilment of the requirements for the degree of

Master of technology (B. tech & M.tech dual degree)

By

Sanjeev Kumar Patra

(Roll Number-711MM1087)

under the supervision of

Professor Sudipta Sen

Department of Metallurgical and Materials Engineering

National Institute of Technology Rourkela

Odisha, India

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Department of Metallurgical and Materials Engineering

National Institute of Technology Rourkela

Rourkela-769008, Odisha, India

CERTIFICATE

This is to certify that the thesis entitled "**Characterization of dual matrix structured ductile iron** " being submitted by **Mr**. **Sanjeev Kumar Patra** to the National Institute of Technology Rourkela, for the award of the degree of **Masters of Technology (B.Tech & M.Tech dual degree)** is a record of bonafide research work carried out under my supervision and guidance. The results presented in this thesis have not been submitted elsewhere for the award of any other degree or diploma. This work in my opinion has reached the standard of fulfilling the requirements for the award of the degree of **Masters of Technology (dual degree)** in accordance with the regulations of institute.

Date: ------------------------------

Dr. Sudipta Sen

(Supervisor)

Associate Professor

Department of Metallurgical & Materials Engineering

National Institute of Technology, Rourkela

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Sanjeev Kumar Patra

Date:

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ABSTRACT

The dual matrix structured ductile iron consists of a soft phase (ferrite) and a hard phase (martensite). The combination of soft and hard phase increases the toughness and strength significantly and meets the requirement of automotive industries for their application in suspension parts. This investigation relates the variation of mechanical properties of dual matrix structured ductile iron with different ferrite and martensite volume fraction as the microstructure and amount of distribution of these phases control the mechanical properties. The DMS ductile iron was obtained by heating the as cast ductile iron to intercritical austenizing temperature of 800°C then holding for 2 mins and followed by quenching in mineral oil. The ferrite and martensite volume fraction was calculated by image analysing software. The effect of ferrite and martensite volume fraction on tensile strength, yield strength and percentage elongation was studied. In this investigation it was found that the Ultimate tensile strength increases and ductility decreases with the increase in martensite volume fraction and decrease in ferrite volume fraction. The dual matrix structured ductile iron with higher volume fraction of martensite results in higher tensile strength and hardness and reduction in ductility. And subsequently the DMS ductile iron with higher ferrite volume fraction results increase of ductility and decrease of hardness and tensile strength. The ultimate tensile stress of DMS ductile iron is much higher than ferritic grades and ductility is somewhat lower than ferritic grade of ductile iron.

Keywords: Ductile iron; Intercritical annealing; Dual matrix structure; Martensite volume fraction

Chapter-01

Introduction

1. INTRODUCTION TO CAST IRON FAMILY:

All cast irons are not brittle as sg cast iron is ductile but a mistakenly consideration of people that cast irons are brittle which is not true. Sg cast iron are the most promising ductility cast iron where carbon is present in its free form as nodules of graphite. It is the only member of cast iron family which has ductility, higher impact resistance as well as the fatigue resistance due to the presence of nodular graphite. The alloy of iron and carbon in which carbon percentage exceeds 2 % are called cast iron. And the cast iron in which carbon is present in form of nodules or spheroids of graphite are called nodular or spheroidal cast iron or commonly known as ductile iron. Speroidal cast iron is the cast iron in which graphite is present in the form nodules or speroids hence called as nodular or speroidal cast iron. SG cast iron as it is ductile gives engineers a combination of toughness,tensile strength,wear resistance and corrosion resistance. Ductile cast iron was first announced in 1948 at American Foundry men's Society Annual Conference which gave a new life to the cast iron family of brittleness to ductility. The spheroids or nodules of sg iron are embedded in ferrite or pearlite matrix. . SG cast iron is produced by addition of a small amount of mg or and cerium in melt or before the melting is done. The mechanical properties of sg iron are better than the grey iron because the presence of graphite in sg iron as nodules makes the ductile iron toughter as it acts as crack arrester unlike the flake graphite in grey cast iron. The mechanical properties of cast iron are mainly controlled by the matrix phase of cast iron and the microstructure of the matrix phase. The shape of the graphite nodules present influences on the range of properties of cast iron. As the properties of the cast iron are basically controlled by its matrix phase so the variety of cast irons are produced hence cast iron is not a single material but a family of material. The final microstructure of matrix phase can be modified by various heat treatment process to get desired properties. The various heat treatment process which can be applied to alter the micro structure are intercriticaly austenizing then quenching gives dual matrix of ferrite and martensite, tempering of DMS gives tempered martensite and the austempering process gives the ausferritic matrices. The application of ductile iron in steering knuckles and brake callipers of automobiles industry and use of sg iron in containers for the storage and transportation of nuclear wastes are the symbols of extensive use of the material in critical application. The cost advantage in production of sg iron and range of properties of the material lead to the increasing application hence production since last five decades .[1-2]

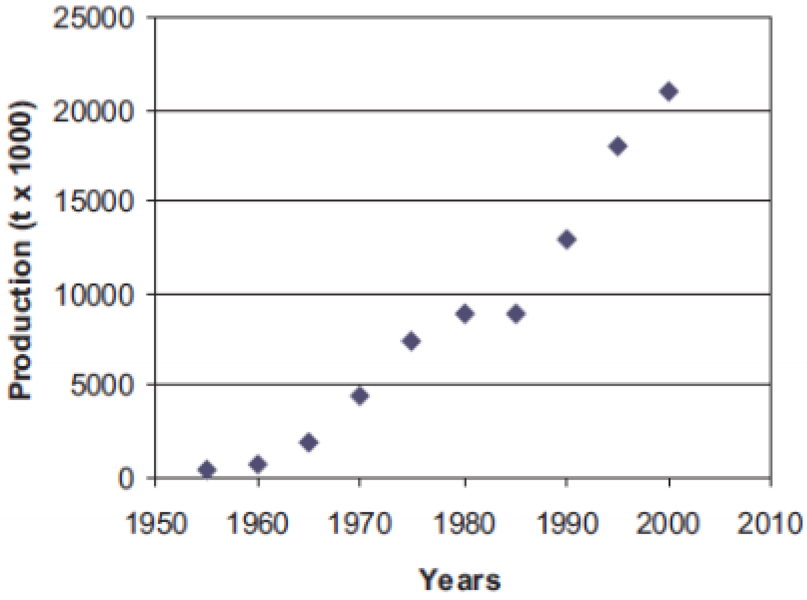


Figure: 1.1 The production of SG iron from 1950 to 2010[25]

The requirement high strength with good ductility in automobile industry for the application in suspension parts are met by the dual matrix structure ductile iron where the matrix phase consists of a soft phase(ferrite) and a hard phase (martensite). Dual matrix structure sg cast iron is a particular kind of sg cast iron which is produced by a special heat treatment process invovles intercritically austenizing and followed by rapid quenching. The intercritical range of temperature is from lower critical temperature to upper critical temperature where ferrite starts to transform austenite and all three phases ferrite, austenite and graphite present. [3]

The comparatively new cast iron with dual matrix structure of ferrite and martensite makes the sg iron a unique combination of ductility and high strength so that it has been used in the some parts of automobiles e.g. suspension parts where high ductility as well as the toughness are required. The dual matrix structure ductile iron is generally exhibits excellent tensile and yield strength with good hardness as pearlitic grade shows and ductility can be compared with ferritic grades[4]

1.3 Objective of the present investigation:

The as cast ductile iron was transformed to Dual Matrix Structured Ductile Iron by a critical heat treatment process named intercritical austenization temperature heat treatment. It is followed by cooling faster than critical temperature to above martensite starts temperature to obtain dual matrix structured ductile iron.

The objectives of the present investigation are:

1. To study the effect of chemical composition on mechanical properties of DMS ductile iron.
2. To study the effect martensite and ferrite volume fraction on mechanical properties of the dual matrix structured ductile iron. And the influence of nodule counts and the nodularity effect on mechanical properties.
3. To study the fractography of dual matrix structure ductile iron with various martensite and ferrite volume fraction.

1.4 Thesis Outline

This thesis comprises of five chapters. The **1st Chapter, 'Introduction',** highlights the objectives of the work carried out and briefly describes about the motivation. The **2nd Chapter, 'Literature Review',** describes review of the past work carried out by the researchers and their results.The **3rd Chapter, 'Experimental Details',** explains the methodology of the working procedure. The application and specification of the equipment has been written . In the **4th Chapter, 'Results & Discussions',** results obtained were analysed and presented in the form of optical images, SEM images, and graphs etc. The experimental results have been presented and their results have been discussed and compared. In **5th Chapter, 'Conclusions'**, the conclusion has been done on the basis of results finding.

CHAPTER-02

Literature Review

2.1 Family of cast iron and background studies:

The alloy of iron and carbon where carbon percentage is more than 2% are called cast iron as casting is the only suitable process to shape this alloy cast. During solidification of the alloy an eutectic reaction takes place hence is an eutectiferous iron-carbon alloy. The castability of an eutectic alloy is best so is the castability of cast iron. Cast iron containing carbon percentage between 2.11 and 4.3 are known as hypo-eutectic cast irons; the cast iron of exactly 4.3%Carbon is called eutectic cast iron; and the iron-carbon alloy more than 4.3% carbon and less than 6.67% carbon are known as hyper-eutectic cast irons.

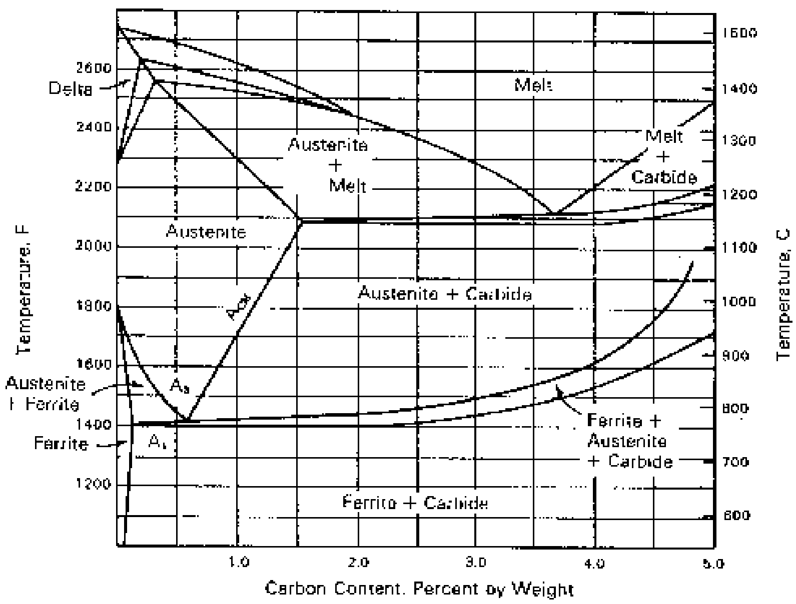


Figure-2.1The iron- iron carbide-silicon ternary diagram sectioned at 2 per cent silicon[26]

The mechanical properties of the cast iron are basically controlled by the shape and form of carbon present. The common cast irons are brittle however sg cast iron are ductile. The production of sg iron in 1948 by adding magnesium changed the perspective of brittleness in all cast iron. [14,15]

2.2 Discovery of Ductile Cast Iron:

As the cost of production of cast iron is relatively low so the researchers were attempting to make cast iron that are comparable to steel in mechanical properties. They were attempting to produce a cast iron with higher ductility.in 1943 Keith Dwight Millis added magnesium to cast iron, he found that solidified castings had no flakes rather sphere of graphites were obtained. After Five years, Henton Morrogh of the British Cast Iron Research Association announced the production of spherical graphite by addition of cerium. Patent 2,486,760 was granted to the International Nickel Company, assigned to Keith D Millis, Albert P. Gegnebin and Norman B. Pilling On October 25, 1949 [5,9,15]

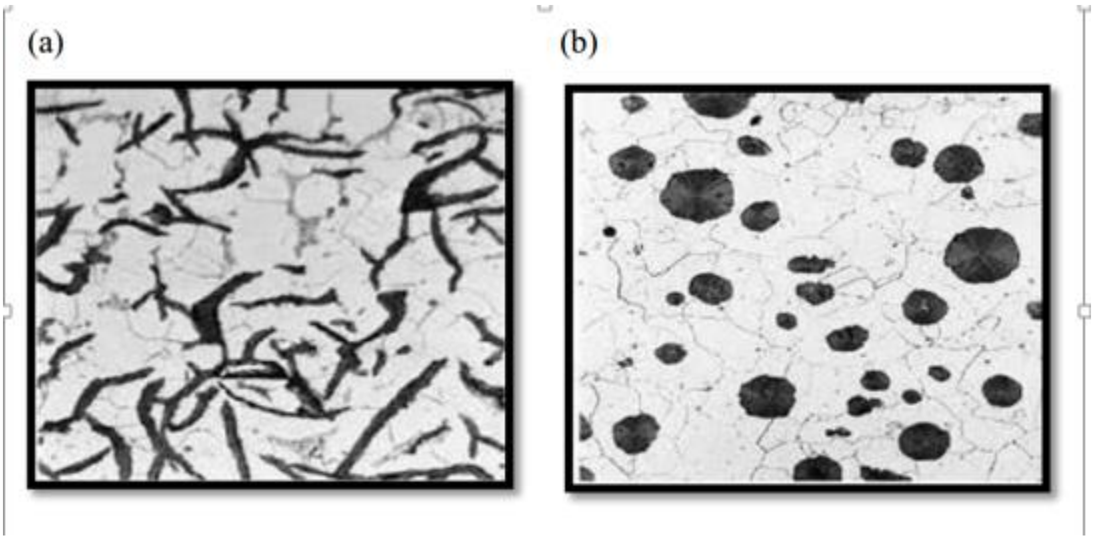


Fig-2.2 Micrstructure of gray cast iron and spheroidal graphite iron (a) gray cast iron, (b) ductile iron

2.2.1 Production of SG Iron:

Si plays an important role in production of sg iron which helps the graphite to form. Removal sulphur and oxygen is done by addition of Magnesium which tie up with them and forms MgS and MgO respectively. MgO floats on the surface and can be separated out hence the oxygen level decreases drastically from about 90-135ppm to 15-35 ppm. MgS also floats on the surface of the melt which is less stable than the MgO and can be separated out. For additional deoxidation the silicon is again added in the form of ferrosilicon. Cerium can also be used to form compound of oxygen and sulphur(oxides and sulphides) which are more stable than the Magnesium oxide and magnesium sulphide and cerium is less volatile than magnesium. Presnece of sulphur makes the graphite places hence are must be removed. Desulphurising agents like calcium carbide are used to combine the sulphur. Proper combination of alloying elements should be added to the melt in order to adjust the nucleation effects, deoxidation and graphitization. G. F. Fisher has given a method to produce Spheroidal Graphite cast iron is given in figure 2.3 [6]

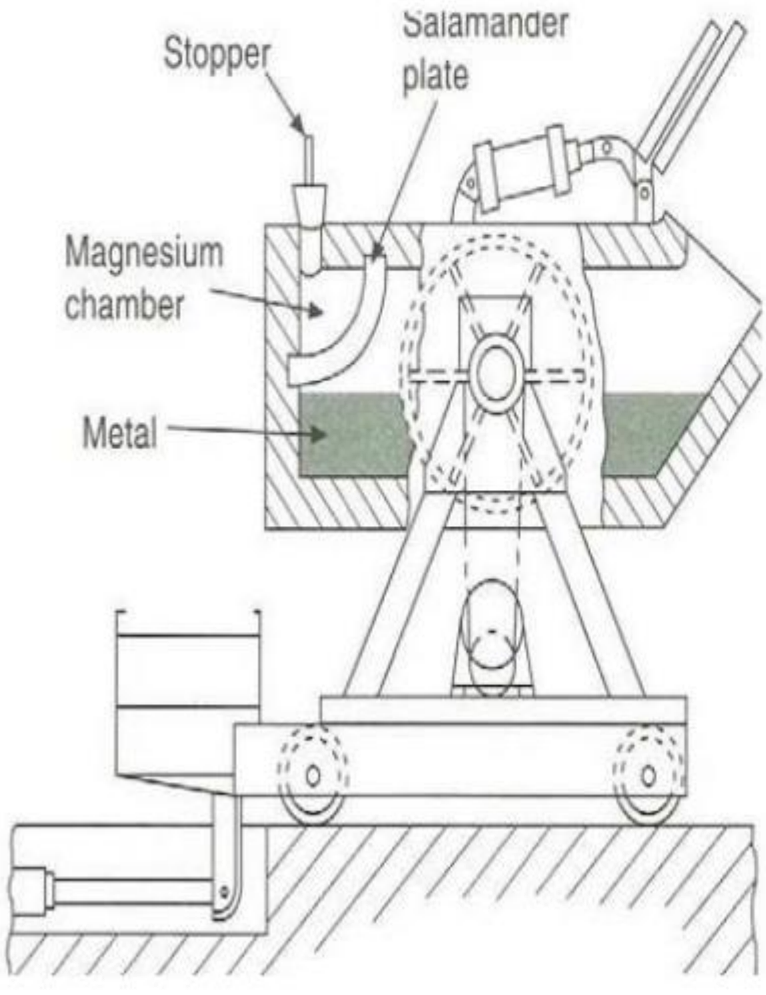


Figure -2.3 G.F. Fisher Method for production of ductile iron [7]

2.2.2 Properties of Ductile Cast Iron:

SG iron has high strength through control of matrix phase microstructure in some cases the strength of sg iron is greater than steel. Ductility of ductile iron is good specially the ferritic grade of this kind. The castability of SG iron is excellent as eutectic reaction takes place during solidification hence it is Foundry men friendly. The machinability of SG iron is excellent as compared to steel. The density of cast iron is lower than the steel. The damping characteristic of SG Iron is better than that of steel. In corrosive atmosphere the corrosion resistance is good.

Materials → **Mechanical properties ↓**	**Ductile Iron** **ASTM A395**	**Cast Iron** **ASTM A48 Class 25**	**Malleable** **ASTM** **A47Grade 32510**	**Cast Steel** **ASTM** **216Grade WCB**
Tensile Strength, Min. psi	**60,000**	**25,000**	**50,000**	**70,000**
Yield Strength, Min. psi	**40,000**	**_**	**32,500**	**36,000**
Elongation, Min. in2"	**18%**	**_**	**10%**	**22%**

Two researchers namely Siefer and Orths after statistical studies of many ductile cast iron found a relation of mechanical properties as given below

$$(Tensile\ strength)^2 \times (Elongation\ \%) \div 1000 = Q$$

Where Q=Constant

The above equation implies that higher the value of Q higher will be the combination of tensile strength and percentage elongation and hence higher would be the performance of alloy.[16,17]

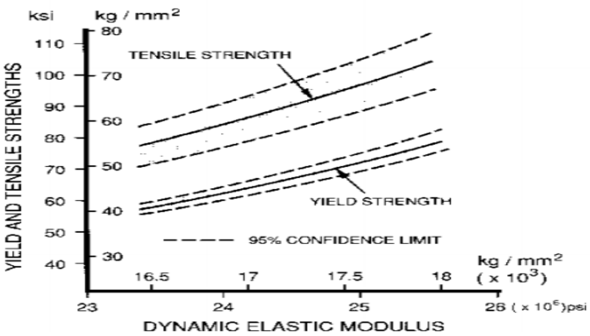


Figure-2.4 Relationships between yield and tensile strengths and dynamic elastic modulus for ductile iron.

2.2.3 Advantages of Ductile Cast Iron:

The requirement of combination of various properties are achieved by the sg iron when other kind of cast iron fails. SG iron generally provides the best combination of ductility and strength to the designers. It ranges the ductility of more than about 18% elongation and high strength of tensile strength of about 850Mpa whereas the Austempered Ductile Iron provides more than 1600MPa of tensile strength.[19]

2.2.4 DIFFERENT HEAT TREATMENT INVOLVES FOR S.G.CAST IRON:

In as cast condition the desired mechanical properties are not readily available as the matrix structure consists generally of ferritic or pearlitic microstructure depending on melt composition. The matrix phase microstructure is basically controlled by the heat treatment applied to the as cast ductile iron. The first step in heat treatment process is the austenising the alloy followed by the slow cooling, fast cooling or moderate cooling can be done to achieve the final microstructure. The microstructure of ferritic grade spheroidal graphite cast iron consists of the ferrite phase which is body centre cubic crystal structure. The austenitic grade of cast iron consist of a metastable face centre cubic crystal structure which reduces the ductile brittle transition temperature. The ferritic grade of spheroidal graphite cast iron is obtained by the annealing process. The austenitic grade of cast iron is obtained by the austempering heat treatment process which is an isothermal heat treatment process resulting in Austempered Ductile Iron(ADI).

Stress relief annealing is generally employed in industrial practice to reduce the internal stress which results in less chance of crack of distortion due to internal stress. This heat treatment process does not involves in the microstructural changes. In some selective heat treatment processes the phase transformation takes place hence the microstructure also.

2.2.5 Austenising the spheroidal graphite ductile iron :

The main aim of austenising the ductile cast iron is that the austenite is the parent phase from which any phase can be obtained depending upon the rate of cooling. The ductile iron of hypereutectic composition is needed to heat up to upper critical temperature in the two phase region of graphite and austenite.

2.2.6 Annealing treatment for the ductile cast iron:

Annealing heat treatment is employed for ductile iron to get the ferritic matrix phase that has high ductility and low strength compared to other grades of cast iron. After austenisation the ductile iron is cooled slowly which results in ferrite microstructure. Pearlitic microstructure is obtained by this heat treatment process and the fine pearlitic microstructure is obtained if air cooling is done and course pearlite is obtained in furnace cooling.

2.2.7 Austempered SG CAT IRON(Autempered Ductile Iron):

Austempered Ductile Iron is a spheroidal graphite cast iron which is produced by an isothermal heat treatment process called austempering.

2.2.8 Austempering:

Austempering is a hardenening treatment process which transforms the austenite into lower bainite in isothermal cooling so that the alloy has lower tendency of distortion and cracks. The alloy is heated to austenized temperature range then cooled in molten salt bath held above martensite starts temperature(Ms) so that the austenite can be converted to lower bainite[18]

The time temperature transformation (TTT) diagram in figure 2.5 shows the phase transformation mechanism.

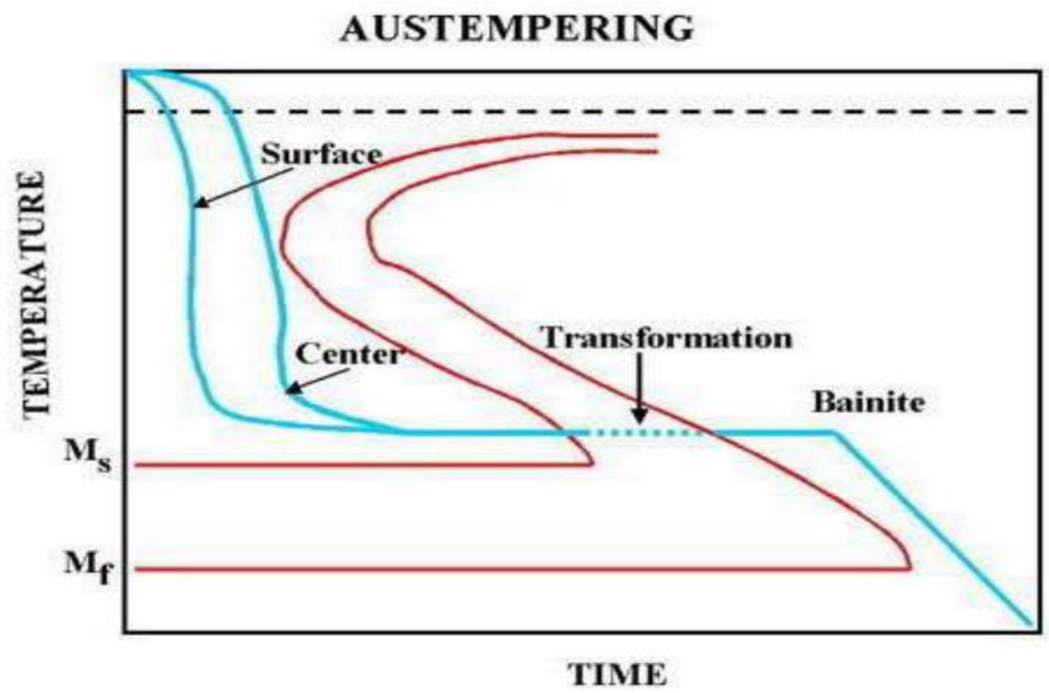


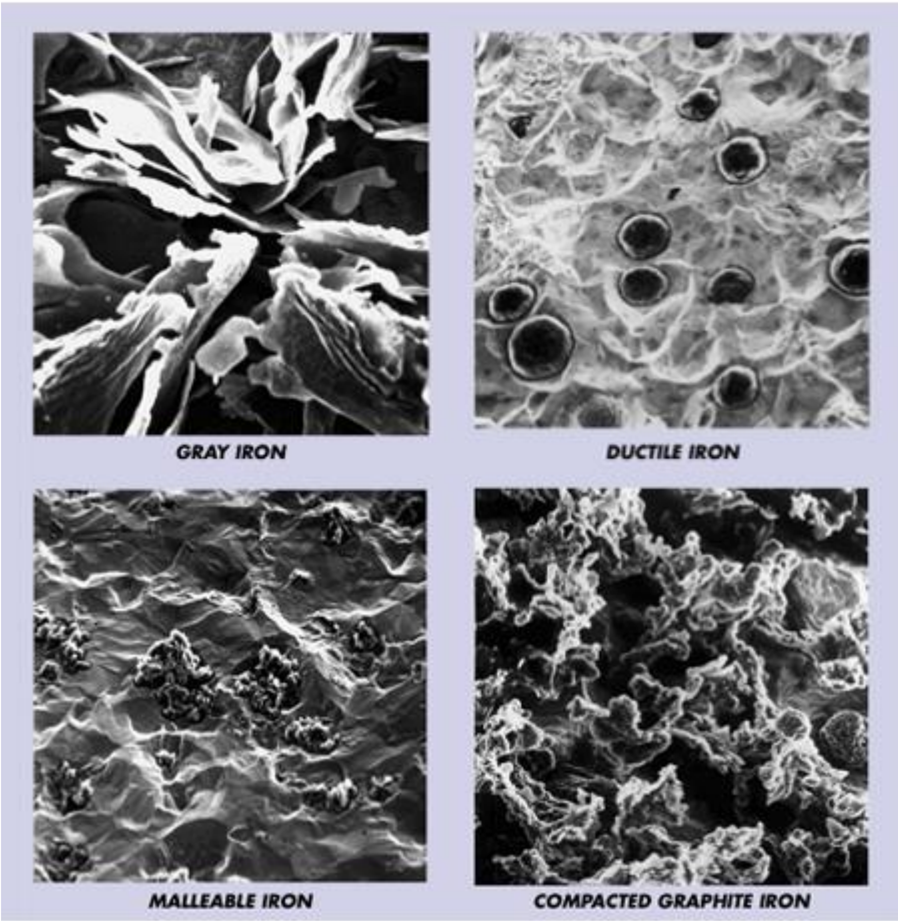
Figure-2.5 TTT diagram for the austempering of Fe-C alloy system

The ADI has various advantages like it has improved ductility without hampering the hardness of the alloy. Less susceptible to distortion and crack. Tempering heat treatment process is not needed which is used to reduce brittleness. Austempered Ductile Iron is has improved impact strength and high endurance limit compared to other grades of cast iron.

2.3 Various forms of graphite in cast iron:

The microstructure of various grades of cast iron such as: ductile iron, gray cast iron , malleable cast iron and compacted graphite iron is given below figure-2.6 Nodularity is clearly observed in ductile iron.

(a) (b)



(c) (d)

Figure- 2.6. These scanning electron microscope (SEM) views illustrate the various forms of graphite found in the cast iron family[25,26]

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2.3.1 Factors that affect the properties of the ductile iron:

Ductile iron possess high ultimate tensile strength and yield strength and its combination with ductility makes the ductile iron a suitable material for the application in heavy engineering industries. The strength and ductility is controlled by its matrix phase and its morphology. The graphite shape also plays an important role in mechanical properties of ductile iron.

2.3.2 Effect of graphite shape:

The huge difference in mechanical properties of gray cast iron and ductile iron is basically due to the presence of graphite in ductile iron as nodules and in gray cast iron as flakes. The shape of the graphite nodules, the nodularity percentage and the distribution of graphite nodules throughout the matrix are the key factors for the performance of ductile iron.

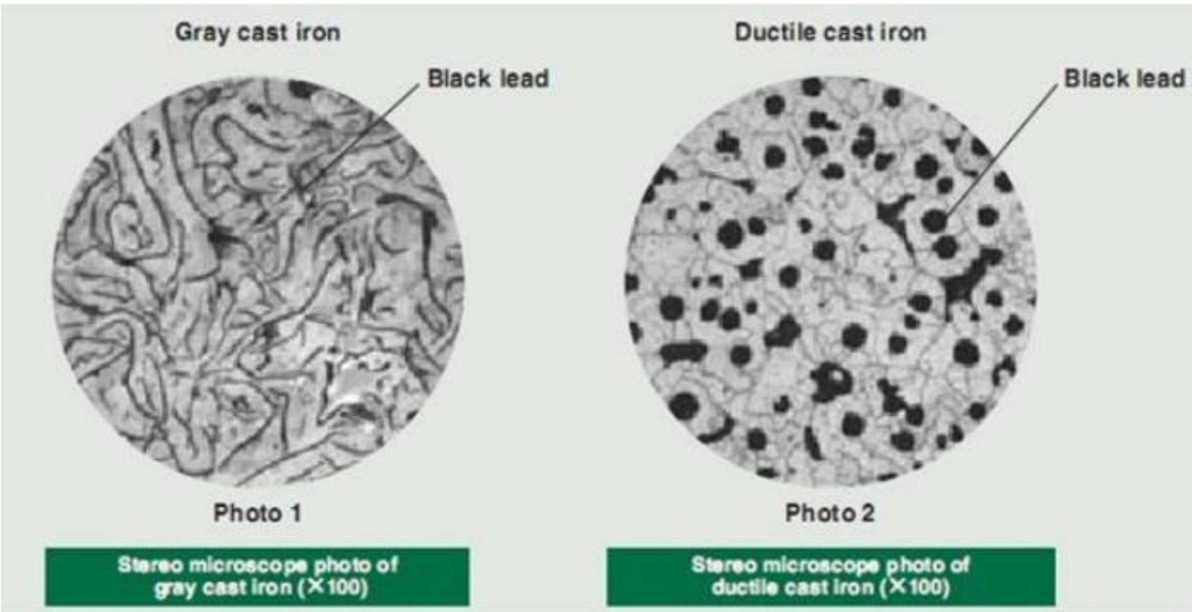


Figure-2.7 microstructure ductile cast iron (a) gray cast iron, (b) ductile cast iron[8]

2.3.3 The Influence of Nodule Count on mechanical properties:

The number of graphite nodules present in 1mm2 area is called nodule counts. The mechanical properties of ductile iron is not strongly influenced by the nodules counts as graphite shape but its effect is always on properties. The nodules count alter the microstructure of the matrix phase which results in change in mechanical properties. In case of as cast ductile iron, with increasing nodule counts decreases the pearlite contents hence tensile strength decreases and ductility increases. Nodule counts plays an important role in size of grains of matrix. With increase in nodule counts the grains of finer microstructure is obtained. Hence by increasing the nodule counts the segregation of carbides can be avoided. [28]

2.4 Modes of fracture in SG Iron:

In Spheroidal Graphite cast iron the mode of fracture is mixed kind where both ductile and brittle fracture is involved. Ductile fracture is the fracture where gross plastic deformation is involved hence is a slow process. Ductile fracture occurs by a slow tearing of metal by expenditure of significant amount of energy. Ductile fracture in nodular or spheroidal graphite cast iron occurs by tearing at the grain boundaries. Brittle fracture involves with little or no plastic deformation and the rate of crack propagation is very high. The brittle fracture in spheroidal graphite cast iron occurs at the grain boundaries or through the grains of matrix phase where graphite nodule is absent. The fracture path goes through the grains is very fast and the fracture surface appears brighter as the cleavage facets reflects the light more. Transition temperature is the temperature below which the ductile material becomes brittle. Almost all the ferrous metal or alloy except some austenitic grade undergo the transition from ductile to brittle. Transition temperature can be raised if the strain rate is increased and the presence of notch raised the transition temperature.[8,10]

2.5 Dual Matrix Structured Ductile Iron:

DMS was first introduced as Soft Eye and Hard Eye in early 80's. The heat treatment applied to get the soft phase of ferrite and hard phase of martensite or bainite generally involves the intercritically austenizing the SG iron into two phase region(γ+α).The alloy is then subjected to rapid cooling faster than the critical temperature to transform martensite from austenite. Intercritical temperature is the area in the Fe-C-Si phase diagram bounded by the upper and lower critical temperature where all the three phases of ferrite, austenite and graphite coexist.[11,12]

2.5.1. Intercritical Austenitizing Temperature (ICAT):

ICAT is the temperature range where the ferrite is partially converted to austenite. It is the temperature range of Fe-C-Si phase diagram bounded by the region of upper critical temperature and lower critical temperature range. Inter critical Austenizing Temperature is the range of temperature where all three phases of ferrite, austenite and graphite are present. The austenite is later converted to martensite when cooled faster than the critical temperature hence the volume fraction of ferrite and martensite is dependent on Inter critical austenizing temperature. The increase of ICAT increases the Austenite volume fraction and hence the increase in martensite volume fraction in spheroidal graphite cast iron. Various studies has shown that the carbon content in austenite is increased by increasing the ICAT temperature and the extra carbon diffuses to austenite from the graphite nodules.[1,5,21-23]

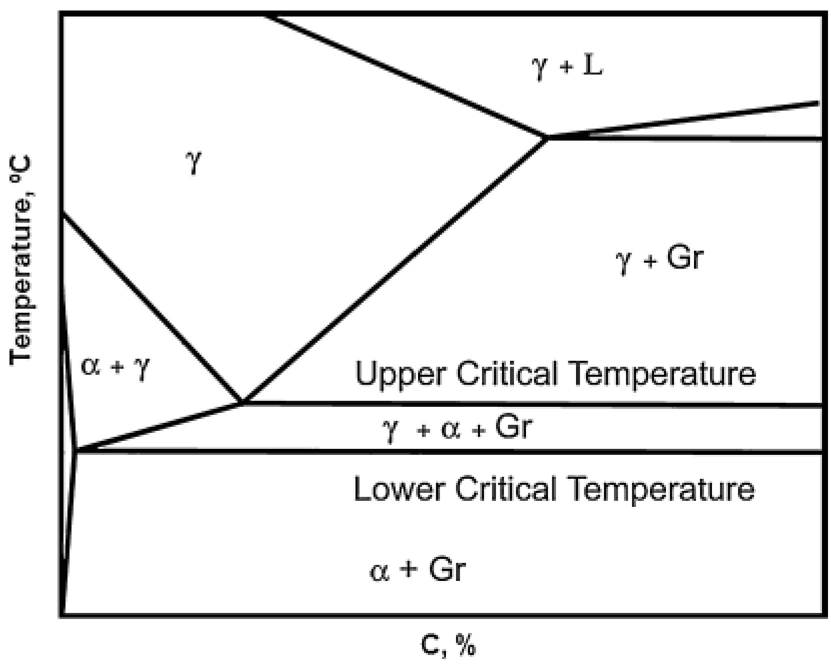


Figure-2.8 Qualitative binary cut of the Fe–C–Si stable equilibrium diagram (2.5%Si).[13]

When starting material for the production of dual matrix structure cast iron is ferrite-pearlite matrix then the carbon diffusion takes place from pearlite to austenite and the carbon from graphite nodules is less there.[27-29]

2.5.2 Austenite formation from ferrite at ICAT:

The grain boundaries of ferrite grains are the most preferable nucleation sites of austenite. The carbides present on the ferrite gran boundaries are more preferential site of austenite nucleation than the carbide present inside the grains of ferrite.[30]

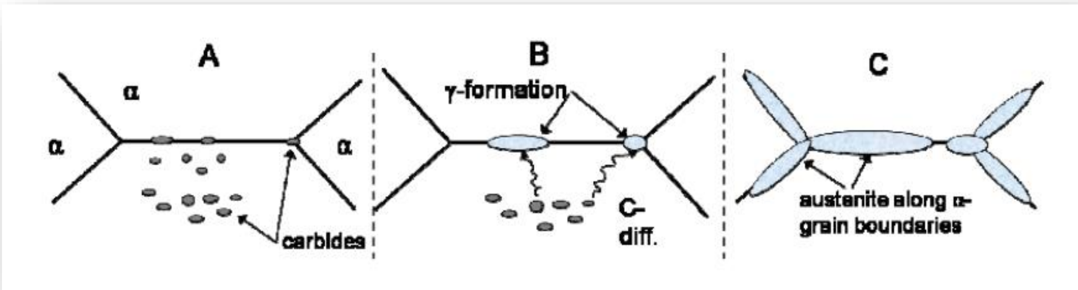


Figure-2.9. Schematic picture of austenite (γ) formation on ferrite-ferrite (α) grain boundaries and triple points.

2.5.3 Quenching process from ICAT:

At ICAT temperature some ferrite microstructure changes to austenite then the alloy is subjected to rapid quenching which is faster than the critical cooling rate to get the martensite out of austenite. The cooling media must be at the temperature below martensite starts temperature which is done by several quenching media e.g. water, oil, salt bath etc. The severity of quenching to get the desired microstructure is controlled by the quenching material used and the bath agitation process. In some complex shapes of materials the bath agitation is required to obtain the uniformity throughout the section and minimization in thermal stress. Higher the severity of quenching in complex shapes the higher would be the internal stress and the cracking may be occurred. [14,31,32]

There are three stages of quenching process such as vapour blanket stage, intermediate contact stage and direct contact stage.

Vapour blanket stage:

On contact with hot material the coolant gets vaporized so that a vapour blanket covers the materials. The cooling rate is very low at this stage as the vapour blanket poor heat conductor.This stage is generally undesirable as the cooling rate is very low at this stage.

Intermediate Contact stage:

In this stage the liquid used as coolant is boiled. The heat is removed very quicly in this stage In this stage the martensite formation takes place as the cooling rate is very fast here.The hardening of steel is dependent on this stage of cooling as the martensite forms rapidly. By controlling the severity of cooling here the retained austenite will be remained in the material.

Direct –contact stage:

In this stage the liquid is cooled down that is heated up in second stage due to the hot material.

The cooling rate is slowest in this stage.[18]

2.5.4 Some quenching medium:

The quenching mediums are used to transform the austenite into martensite. The quenching power of the coolant is decreased as the temperature of the coolants are increased. Some commonly used coolants are water, brine, oils and polymer quenchants.

WATER:

The cooling power of water is optimum when it is heated to about 20-40**°C.** The cooling power of water is higher than brine and less than oil. Quenching by using water is hindered by the formation of vapour hence the agitation must be done. The drawback of water quenching is its formation of oxides with the metal.

BRINE:

Brine is the aqueous solution of sodium chloride present about 10% by weight. Cooling power of brine is less than water so it can be applicable where less severity of quenching is required. Direct contact with the alloy at early stage is possible by using brine as the deposition of crystals on the surface of hot alloy takes place and the disruption of crystals destroys the vapour blanket.

OIL:

By using various types of oil like vegetable oil, mineral oil, animal oil and their blend the variation of cooling power can be obtained which is an advantage. Generally the cooling power of oil is between the water at 40°C and water at 90° C so optimum cooling rate makes it suitable for use. The oil that is having higher viscosity is less volatile so has the more cooling rate. The disadvantages of using oils are it is high cost and is highly inflammable.

2.5.5 Epitaxial Ferrite Formation:

Epitaxial ferrite formation takes place when cooling from the intercritical austenising temperature on the retained ferrite which was present during transformation of ferrite from austenite. Depending on the amount and type of austenite stabilizing elements present resulted in the retransformation of austenite into ferrite. Austenite stabilizing like carbon is the interstitial solute atom which has more diffusivity that some other elements and hence high amount of other elements are required to resist the retransformation. [33, 34]

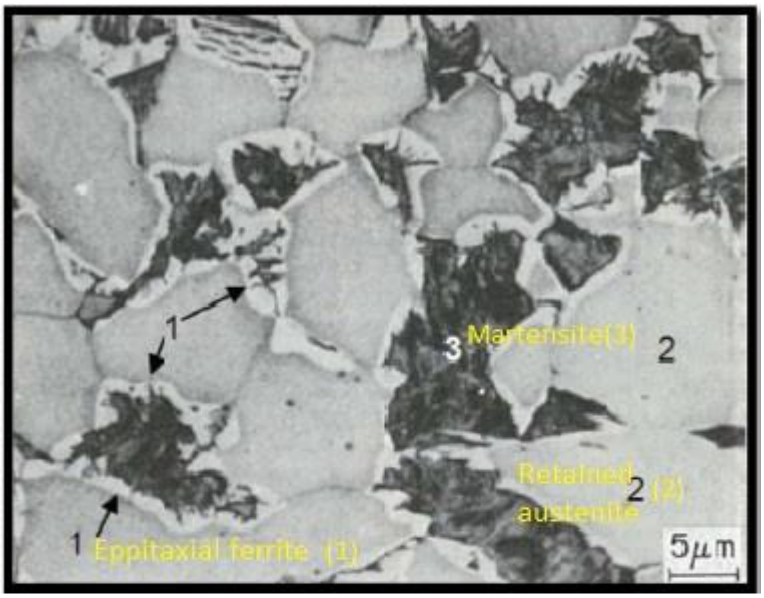


Figure-2.1.1 Eppitaxial ferrite formation at retained austenite. [35]

The formation of epitaxial ferrite takes place on the previously existed ferrite surrounded by austenite pools when subjected to slow cooling. During fast cooling rate the preferred nucleation sites for the epitaxial ferrite are the triple points of the austenite grains. [34-37].

2.6. PHASE TRANSFORMATION TO MARTENSITE:

The austenite to martensite phase transformation occurs when cooled faster than critical cooling rate to below martensite start temperature. Martensite phase transformation reluts with volume expansion and diffusionless transformation. The martensite phase is a body centred tetragonal crystal structure. Martensite is the supersaturated solid solution of carbon in iron. The volume expansion is clearly visible in figure-2.1.2.of dilatation curve. [38]

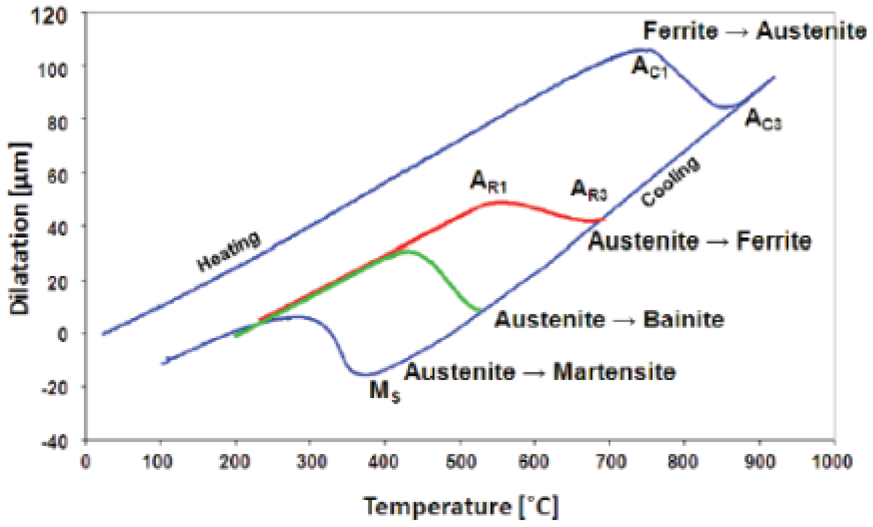


Figure-2.1.2. Dilation curve

Martensite formation involves the plastic deformation of austenite accompanied with diffusionless phase transformation. Martensite transformation from austenite phase involves volume expansion. The dislocation density in ferrite crystal in dual matrix structure cast iron increase with martensite formation. Martensite phase is the supersaturated solid solution of carbon in iron hence it is a unstable phase. [39]

2.6.1 Tempering:

Tempering is the heat treatment process applied to steel and cast iron which were previously quenched to increase ductility and toughness and to reduce the internal stress. The brittleness of the quenched material are reduced by converting the martensite phase to tempered martensite. Heating the martensitic phase to above martensite starts temperature then isothermal cooling is done. In dual matrix structure cast iron, the martensite phase converted to tempered martensite phase which is less brittle.[41-44]

2.7 A Review of past Research work:

M.Moshrefi-Torbati and Ali M.Rashidi, have studied the mechanical properties of DMS SG iron that has been tempering heat treated. For this study they quenched the sample from intercritical austenising temperature and then tempered. For the analysis the sample which were taken was having chemical composition of 3.65% C , 1.33%Ni ,0.29%Mo, 0.017%P, 1.94% si, 0.012% S and 0.28% Mn and the rest percentage is Fe. They austenized sample at 950°C and hold for a duration of 2 hrs followed by quenching process. The influence of various tempering temperature on the mechanical properties e.g. 0.2% yield strength, UTS, impact strength as well as percentage elongation is plotted in figure-2.1.3

And the influence of duration of tempering on mechanical properties at 500C temperature is shown in figure-2.1.4

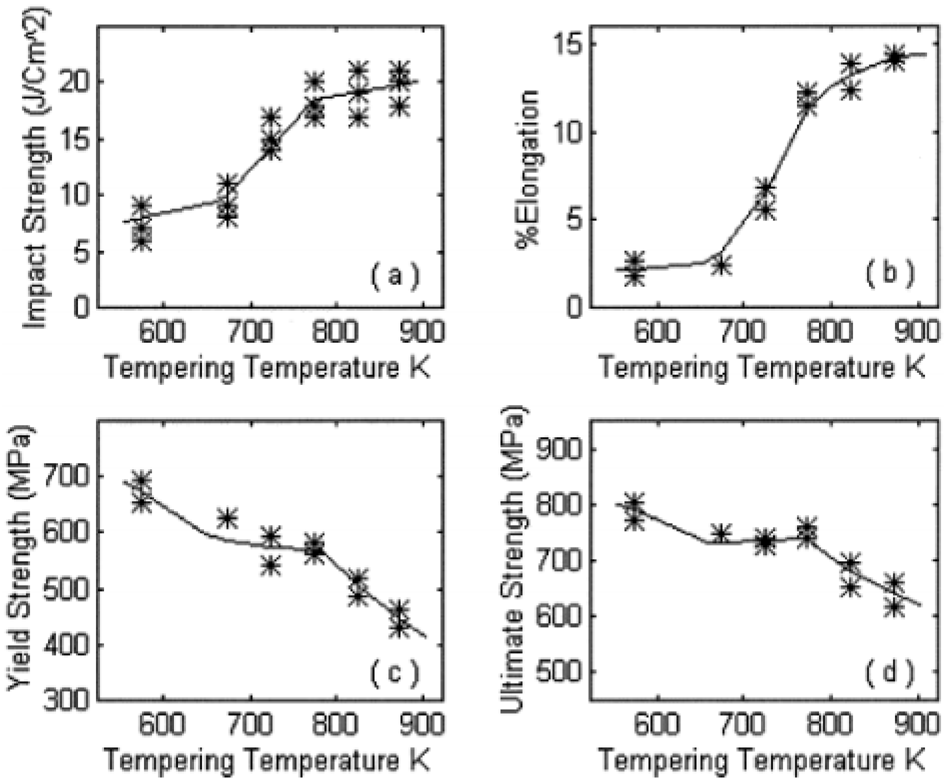


Figure-2.1.3 (a)Impact strength vs tempering temperature, (b)% elongation vs tempering temperature, (c)Yield strength vs tempering temperature, (d)UTS vs Tempering temperature.

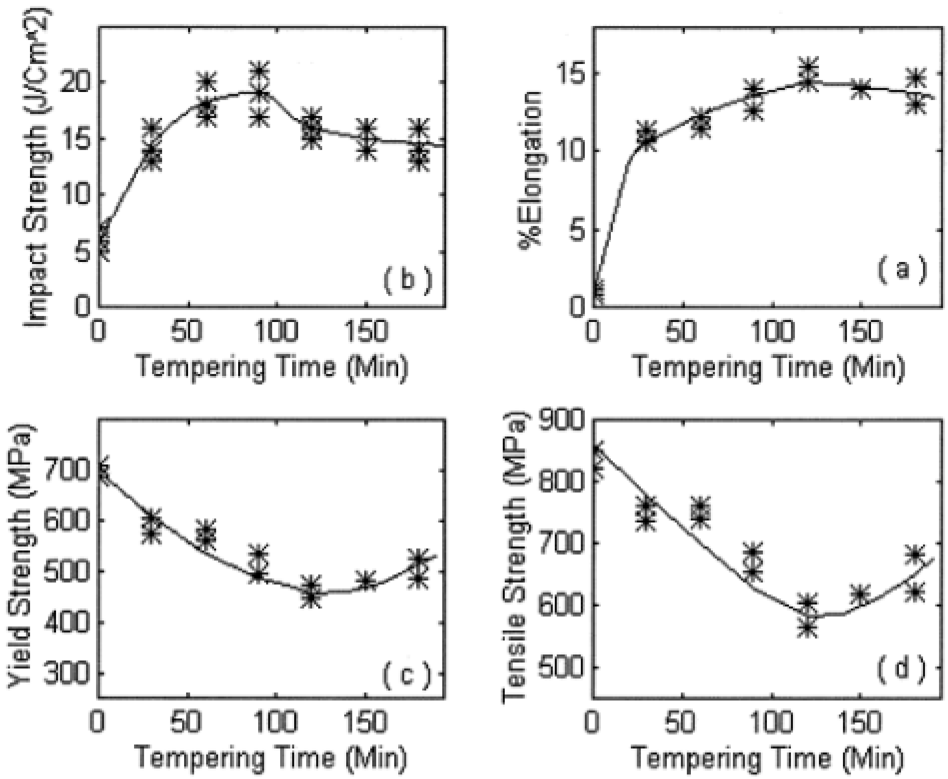


Figure-2.1.4 (a)Impact strength vs tempering time, (b) % elongation vs tempering time, (c) Yield strength vs tempering time, (d)UTS vs tempering time.

They had done the tempering at various temperature of 300°**C**, 400°**C**, 500°**C** and 600°**C** for about 1 hrs. Then the sample was taken for mechanical properties testing like tensile testing after dimensioning the material .They found that with increasing tempering temperature the ductility increases but the increase of ductility becomes slow for the samples which were tempered at temperature between 400°**C**and 500°C. However they also found that with increasing in tempering time the ductility increases dramatically [45]

I.C.Hsui and L.C.Chang in 2005 investigated the erosion characteristic of spheroidal graphite cast iron. They quenched the sample from austenite region then tempered the samples and then studied the behaviour of erosion of the samples which were dual matrix structured ductile iron. The chemical composition of the samples were 2.88%C, 0.29%Mo and other elements in minimum percentage and rest are Fe%. They autenized the sample at 950C for their investigation and then held for about 2hrs and then quenched the samples so that the austenite present in the alloy gets converted to martensite which is a hard phase. As their aim was to produce the lower and upper bainite structure so they austempered the samples at 420°C and 280°C for about 1 hr. After the austenisation of spheroidal cast iron the specimen surface were cleaned to remove the oxide layer present. The specimens were then subjected to erosion wear test and the total weight loss for specific duration was measured. The wear mechanism in erosion test was understood. From this investigation they found that the with impact angle the erosion rate increases initially and then decreased. [46]

In july 2005 Kadir Kocapete et.al investigated the fractography of fracture surface resulted in tensile loading of dual matrix structured spheroidal graphite cast iron which were intercritically austenized and then quenched and tempered. The specimens taken for study were having various martensite and ferrite volume fraction. The chemical composition of the samples taken for the investigation was 3.5%C , 2.63%Si and 0.318 %Mn with other elements and rest are Fe %. The samples of spheroidal graphite cast iron were austenized at temperature of 900°C then quenched in the quenching media oil then tempering is carried out. The mechanical properties were compared between the dual structured cast iron and as cast iron. The study of both the specimens revealed that in the as cast microstructure the ferrite and graphite nodules were present and in the dual matrix structured ductile iron the ferrite, martensite and graphite microstructure is present. After that the DMS samples were tempered and the as cast cast iron were then annealed at different temperature of 795°C and 815°C for the short duration of 30 secs. The mechanical properties were measured like tensile properties, yield strength and percentage elongation and the fractography examination was done in scanning electron microscope. The microstructure of all the specimens were revealed using optical microscope. The ductility of dual matrix structured ductile iron was compared with ferritic grade of ductile iron and they found that the ductility of ferritic grade of ductile iron has more ductility than the DMS ductile iron. In case of DMS ductile cast iron the mode of fracture changes from ductile to moderate ductile fracture. They studied the fractography studies of DMS ductile cast iron and found the mixed fracture mode. The tensile strength and 0.2 % proof strength of the DMS is found to be between the quenched ductile iron and ferritic or pearlitic grade of ductile iron.[24]

In 2006 the research work of Gulcan Toktas et.al on the variation of various mechanical properties and impact toughness with varying the microstructure of matrix phase. They had taken the specimen with chemical composition of 3.6% C, 2.29% Si, 0.011 S %, 0.08Mn% and the rest percentage is Iron. They divided the cast ingot into five groups for this study. One of the group materials were as cast ductile iron and second group was taken for ferritic heat treatment process. The ferritic heat treatment was carried out in two stage process, first stage was the isothermal holding at temperature of 920°C and the second stage isothermal holding was done at temperature of 720°C and it was for 7 hrs and then furnace was allowed to cool to room temperature. All the rest groups of materials were cooled at different rate of cooling like at still air, and some inside furnace of blocked of air. The second group of ductile cast iron was found to fully ferritic matrix embedded in graphite nodules. The microstructure study of as cast ductile iron reveals the ferrite-pearlite matrix embedded with graphite nodules where some graphite spheroids are embedded by ferrite phase. The ferrite envelopes were also surrounded by pearlitic matrix phase. The third group of materials which were cooled at different rate shown nearly same microstructure. The one which was cooled in still air and forced air had same microstructure but the ratio pearlite to ferrite was more in the specimens which was cooled in forced air and in latter case the ferrite was embedded in pearlite phase. The mechanical property tensile strength was influenced by the pearlite content. The SG iron of ferritic grade was found to be the 0.2 % proof strength of 245 Mpa and the pearlitic grade of ductile iron showed the 475 MPa. They found that the ductile iron with pearlitic grades has increased due to increased pearlitic level. The increased pearlitic level ductile iron of ferrite/pearlitic matrix was found to be more tensile strength of about 25%. They found that the the ductility of ferritic matrix structure is more than the pearlitic matrix structure. Hence the fracture energy of ferritic grade ductile iron has highest fracture energy and with increasing the pearlite level in ductile cast iron proportionally reduces the fracture energy of the ductile cast iron. The values of hardness in pearlite matrix structure was found to be increased severely with increasing pearlite level. After fractography studies in FESEM they found that the path of fracture propagation in ferrite/pearlite matrix ductile iron connects more nodule of graphite and avoids the pearlitic phase. The fracture mode of ferrite grade of ductile cast iron was found to be the dimple pattern which is a ductile fracture. And in ferrite/pearlite matrix phase dimple and cleavage pattern of fracture mode was observed. In pearlitic areas the fracture mode was seen to be river pattern of brittle fracture.[40]

J.O. Choi, J.Y. Kim, C.O. Choi, J.K. Kim and P.K. Rohatgi, examined the influence of rare earth metallic elements on the formation microstructure and some mechanical properties like ductility, tensile strength, 0.2% proof strength of thin wall spheroidal graphite cast iron casting. Spheroidal graphite cast iron castings having thickness of 2 mm, 3mm, 4mm, 6mm, 8mm, and 25mm and different proportion of rare earth element was casted in sand casting mold to recognize the effect of specimens thicknesses and the amount of rare earth metal on the formation microstructure and some mechanical properties like tensile strength, ductility etc. The effect of amount of rare earth metal and the thickness of samples on the formation microstructure. They calculated the number of graphite nodules. The shape of graphite nodules were observed, and amount of ferrite and spheroidisation were also observed. From their investigation on effect of rare earth metals on mechanical properties they found that the 0.2% yield strength of the samples which were prepared with RE were less than the samples produced without use of rare earth elements. The ductility was improved by adding the rare earth metals to the metal casting such as by addition of RE up to 0.3 % the ductility increases sharply. The addition of rare earth metals of 0.02 % resulted in the reduction of size of graphite nodules. They found that the number of graphite nodules are decreased by increasing the section size of the casting. In the study it was observed that the chilled zones were present in thickness of about 2 mm size but chilled zone was absent in the section size of more than 2 mm. The spherical shape of graphite is increased when the addition of rare earth metals was between 0.02-0.04 %. By comparing the tensile results of all the samples it was observed that the tensile strength decreases with addition of rare earth metals but the ductility increases with that.[47]

A.Kutsov, Y.Taran, K.Uzlov, A.Krimmel and M.Evsyukov, in 1999 investigated the of kinetics of transformation to bainite subjected to isothermal transformation in Ni-Mn-Cu-Mo alloyed spheroidal graphite cast iron. In the study the bainite(upper and lower) formation and the kinetics of phase transformation was the objective. They found that in upper bainite structure, feathery type morphology is present and in the lower bainite the plate like structure is present. [48]

Chapter-03

Experimental Details

3.1 Introduction:

All the experimental procedures implemented during the course of work has been briefly described in this chapter. The specifications of the equipment and their working details have been reported concisely. The step by step procedure adopted was firstly the heat treatment of ductile iron then their microstructural studies followed by determination of mechanical properties and the fractographic studies are given in the figure-3.1of work plan.

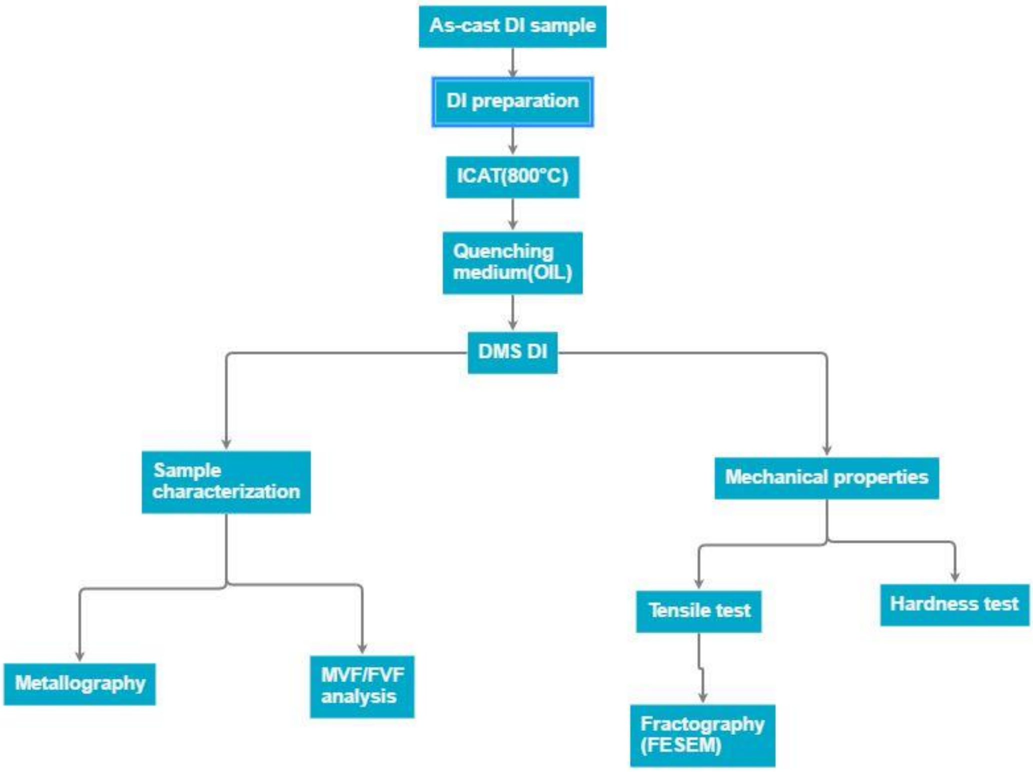


Figure-3.1 Work plan of the project.

3.2 Test Specimen Preparation:

3.2.1 CHEMICAL COMPOSITION:

The chemical composition of all the ductile iron samples taken for the present investigation has been given in Table-3.1. The as cast ductile iron sample was produced at L& T Kansbahal foundry prepared in accordance with ISO 1083. Tundish cover reactor was used to nodulise the graphite.

SL. NO.	%C	%Si	%Mn	%S	%P	%Cr	%Ni	%Mo	%Cu	%Mg	%Ce	%Fe
ACDMS-1	3.45	2.07	0.15	0.008	0.024	0.02	0.15	0.001	0.007	0.043	0.004	94.073
ACDMS-2	3.52	2.04	0.17	0.009	0.022	0.02	0.11	0.001	0.02	0.042	0.007	94.039
ACDMS-3	3.63	2.19	0.25	0.008	0.033	0.03	0.09	0.001	0.014	0.042	0.0007	93.7113

Table-3.1 The chemical composition of as cast ductile iron.

3.3 HEAT TREATMENT

Upon fabrication the specimens were intercritically autenitised at temperature of 800.C in two phase region. The process was subsequently followed by quenching process, where in mineral oil was used as quenchant. The heating and cooling curve is shown in figure-3.2

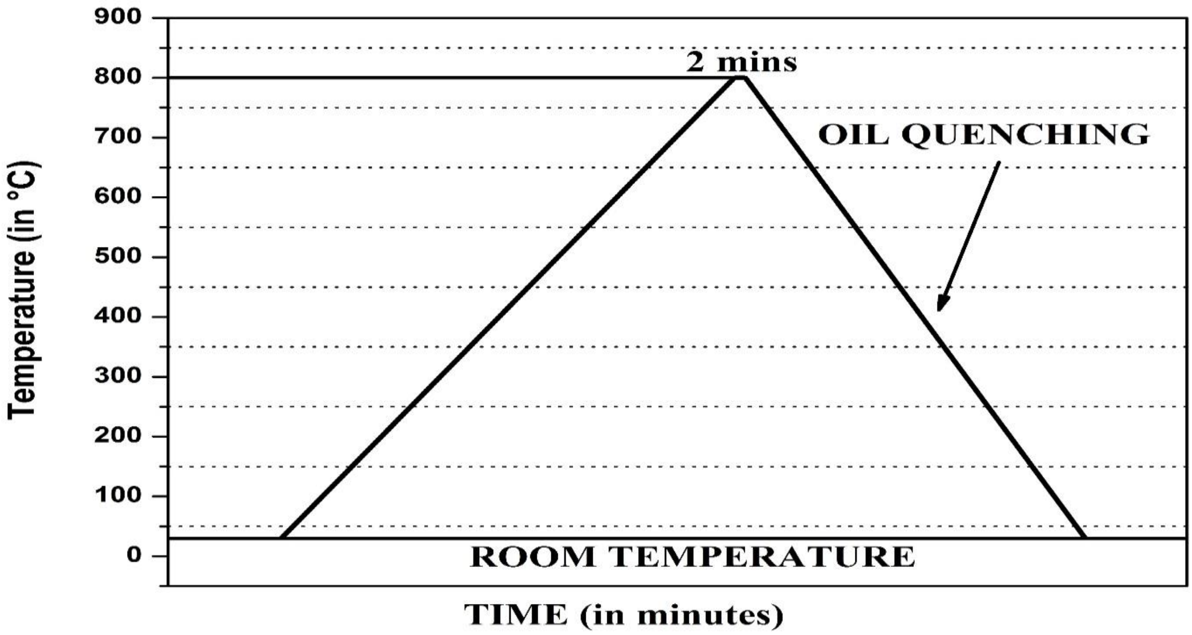


Figure-3.2 Heat treatment curve to obtain DMS ductile iron.

The specimen was coded according to the heat treatment involved and the chemical composition and ferrite martensite volume fraction(%) of the dual matrix structured ductile iron.

Specimen Code	Intercritical austenitizng temperature (ICAT)	Quenching medium	Ferrite volume fraction(%)	Martensite volume fraction(%)
F23M70-DMS	800°C	Oil quenched	23	70
F20M64-DMS	800°C	Oil quenched	20	64
F25M60-DMS	785°C	Oil quenched	25	60

Table-3.3 Description of Specimen codes used in the present studies.

3.4 OPTICAL MICROSCOPE STUDY:

The microstructure of the DMS cast iron was studied by the use of the optical microscope.

Figure-3.3. Metal power image analyser with optical microscope.

3.5 MEAUREMENT OF THE FERRITE AND MARTENSITE VOLUME FRACTION:

By using the analysis software metal power image analyser the martensite and ferrite volume fraction were determined. All the dual matrix structure ductile iron specimens were used to determine their ferritic volume fraction and martensitic volume fraction.

3.6 HARDNESS MEASUREMENT:

Vickers hardness testing machine was used to determine the hardness values of dual matrix structure ductile iron. An indentation load of 30 kgf was applied for dwell time of 10 seconds and the reading were noted. In Vickers hardness, a diamond indenter in the shape of right pyramid with a square based was used.

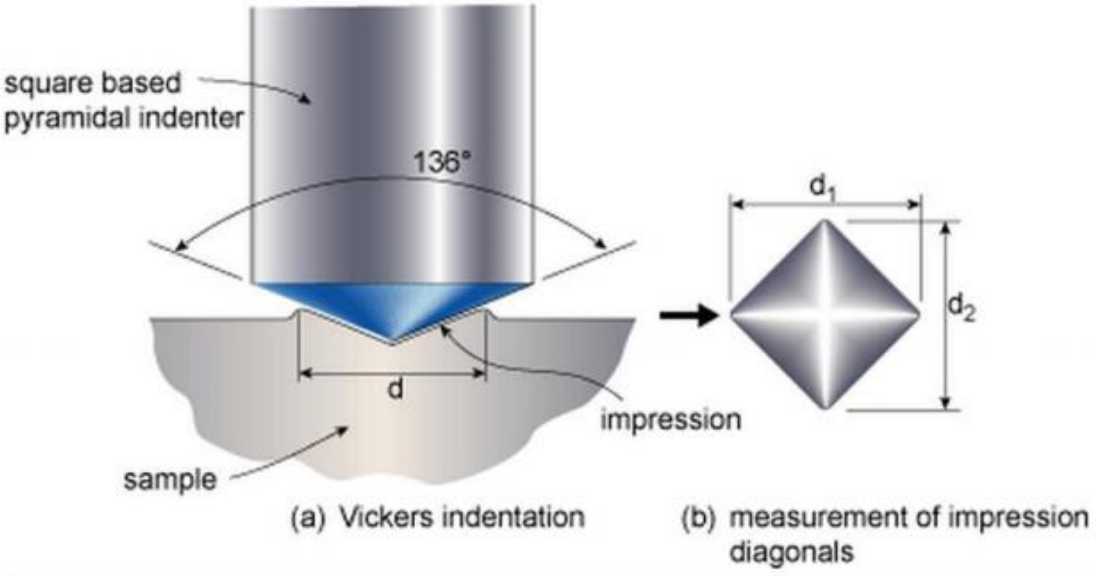


Figure-3.4. Schematic figure of the Vickers pyramid diamond indentation.

An angle of 136° was maintained between opposite faces of the indenter. Generally the results of Vickers hardness test are reported in Kg/cm^2 which equates to load divided by the square of the diagonal of the indentation.

Figure-3.5 Vickers hardness tester.

3.7 TENSILE TESTING:

The tensile test was carried out as per ASTM E-8 Universal Testing Machine. INSTRON-1195 was used to perform the test. A load of 100 KN was applied at a cross head speed of 1mm/m. All the specimens were tested at room temperature.

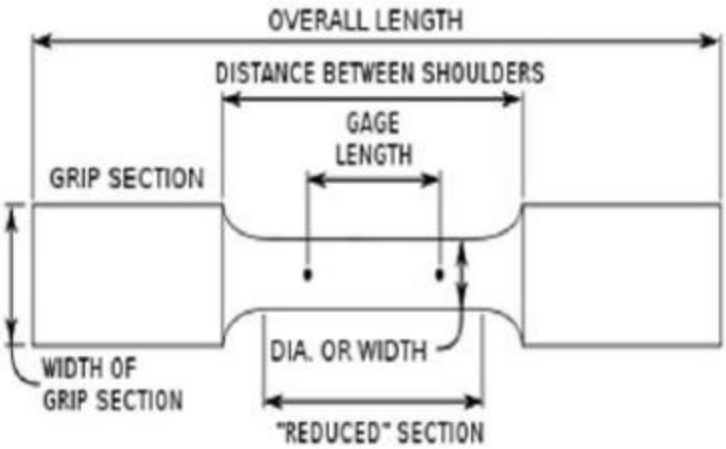


Figure-3.6. Specimen preparation for tensile testing.

Following this testing the load-displacement graph were obtained from which the mechanical properties such as UTS, percentage elongation, yield strength were estimated.

Figure-3.7. INSTRON-1195 machine tensile testing machine.

3.8 IMPACT TESTING:

Impact energy was measured by employing Charpy V-notch test according to ASTM-E23. For the testing the samples were shaped as shown in figure 3.8 and the result shown in the Charpy scale was noted.

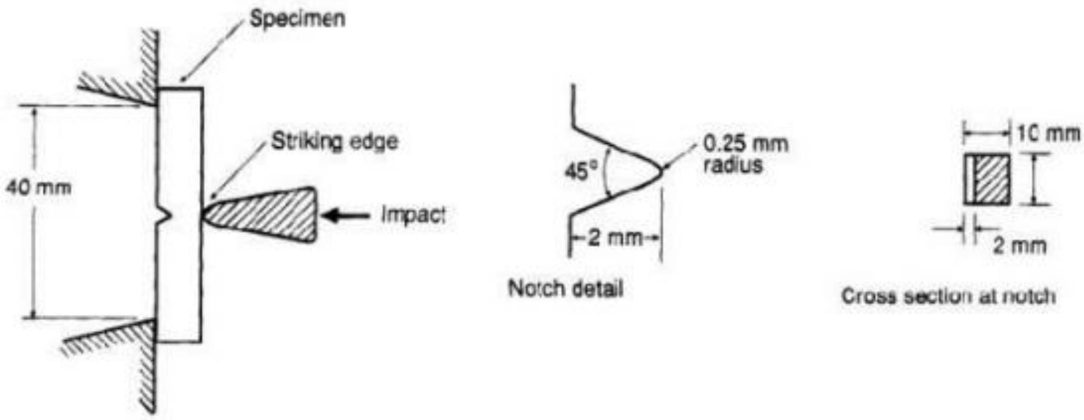


Figure-3.8 Material preparation for Charpy testing.

3.9 FRACTOGRAPHY:

Scanning Electron Microscopy was used to observe the fracture surface of the tested samples.

Fracture surface or surface morphology of the samples which fractures in different manners(ductile, Brittle and mixed mode fracture) after tensile test and impact test were analyzed by using Scanning Electron microscopy (SEM).For this samples were cleaned with Acetone to remove any dust or impurity on the surface of specimens before SEM

Chapter-04

Results and Discussion

4.1 *Microstructures:*

The microstructure of the as spheroidal graphite cast iron (ductile iron) and the microstructure of dual matrix structured ductile iron have been studied in this chapter.

4.1.1 As cast microstructure:

The chemical composition of the as cast dutile iron having ferrite and graphite matrix structured cast iron is given in table 3.1. The microstructure of as cast ductile iron consists of ferrite matrix embedded with graphite nodules.

4.1.2 Dual matrix structured ductile iron:

The as cast ductile iron samples of different composition given in table-3.1 were intercritically austenized at 800°C and held for 2 minutes then followed by rapid quenching in mineral oil. At intercritical temperature the ductile iron remains in three phase region of ferrite, graphite and austenite. Nucleation of austenite takes place at prior ferrite/ferrite grain boundaries. The quenching of the samples from intercritical austenization temperature (ICAT) after holding there for only 2 minutes resulted in dual matrix structured ductile iron of ferrite and martensite matrix.

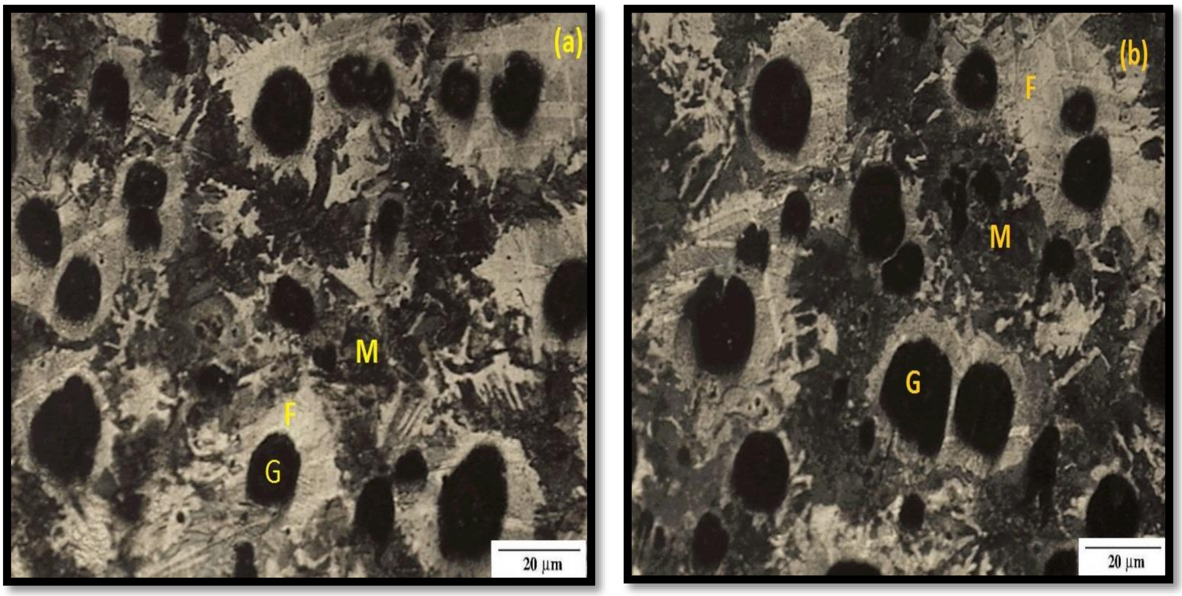


Figure-4.1. Microstructures of dual matrix structured ductile iron at 200X for (a) F23M70-DMS (b) F20M64-DMS, F- Ferrite; M- Martensite, G-graphite nodules.

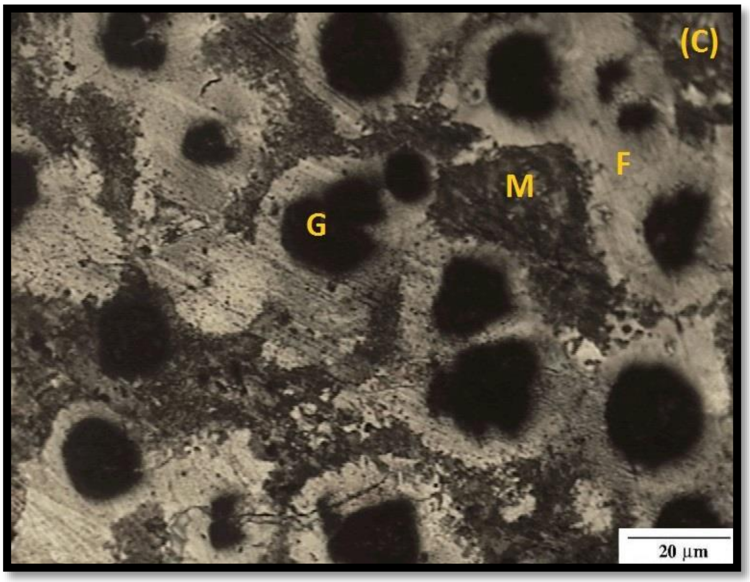


Figure-4.1. Microstructures of dual matrix structured ductile iron at 200X for (c) F25M60-DMS F- Ferrite; M- Martensite, G-graphite nodules.

4.1.3 Infuence of chemical composition on ferrite and martensite volume fraction:

The sample F25M60-DMS having silicon 2.19 % resulted in highest ferrite volume fraction with highest percentage elongation. As silicon is a ferrite stabilizer its presence resulted in restricted transformation to austenite at intercritical austenizing temperature hence less volume fraction of martensite was obtained after quenching. In sample F23M70-DMS with Si 2.04% resulted in lowest percentage elongation as ferrite volume fraction is 23%.

As the ferrite volume fraction of the dual matrix structured ductile iron increases the percentage elongation increases. The ferrite volume fraction represents the ductile fracture of the dual matrix structured ductile iron. The impact energy of DMS ductile iron increases with increase in ferrite volume fraction as it increases the ductility of the materials.

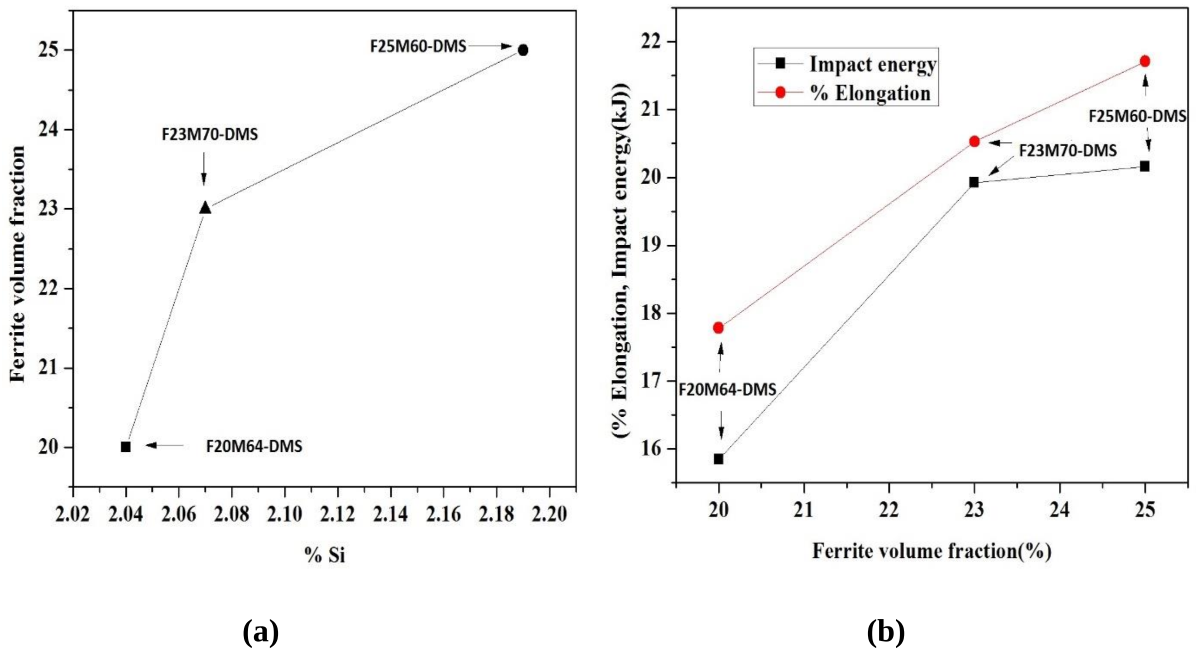


Figure-4.2. (a) Silicon Percentage vs ferrite volume fraction. (b) Percentage elongation vs FVF.

Ni is an austenite promoter hence at intercritical austenizing temperature Ni promotes austenite formation resulted in increase of martensite volume fraction in DMS ductile iron. In the sample F23M70-DMS with 0.15 Ni% resulted in 70 % volume fraction of martensite whereas in F25M60-DMS with 0.09% Ni resulted in 60% volume fraction of martensite. As the mechanical properties are basically controlled by the matrix structure of ductile iron hence with increasing martensite volume fraction the ultimate tensile strength and hardness increases.

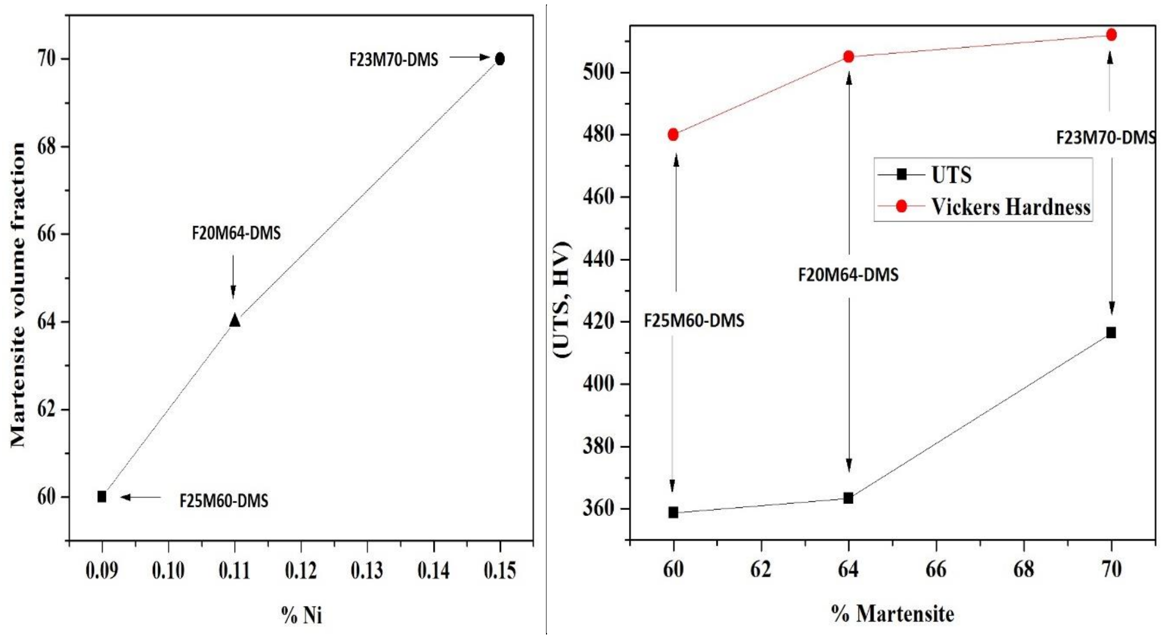


Figure-4.3 (a) martensite volume fraction vs % Ni (b) UTS,HV vs Martensite volume fraction.

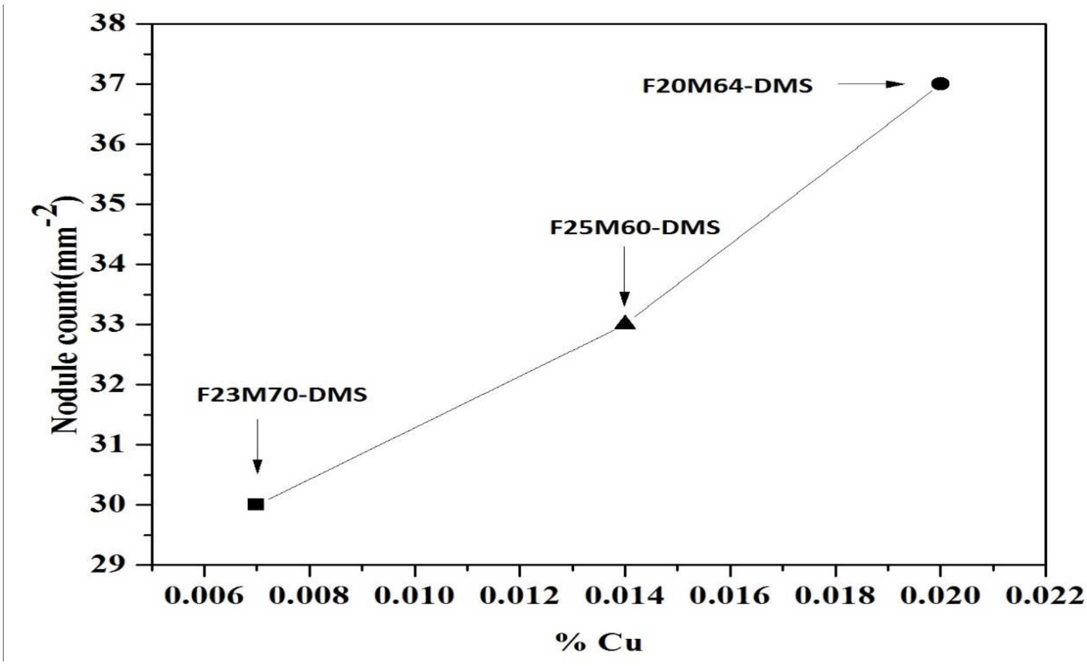


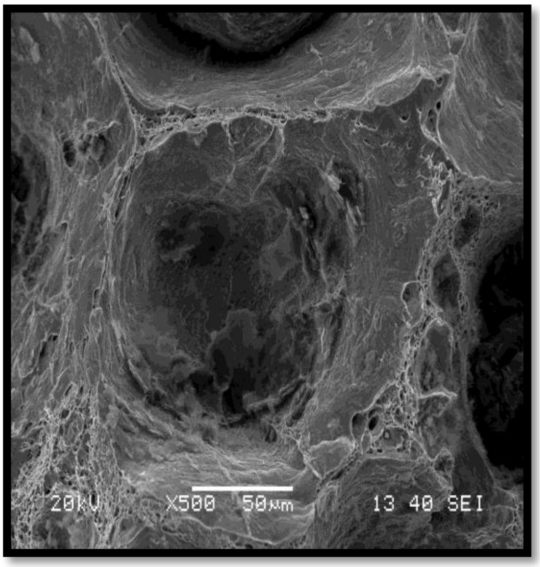
Figure-4.4 Nodule count vs percentage copper.

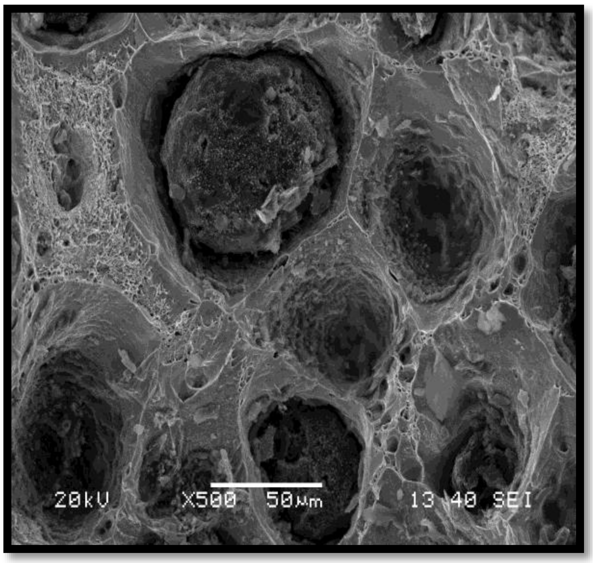
With increase in nodule count the mechanical properties like tensile strength, yield strength increases as shown in figure which is achieved by increasing copper percentage. The magnesium content is the most important element for the morphology of eutectic carbon in cast iron. Also, the size of carbon nodule increases with increasing Cu% contents. Although, the effect of Cu on the size of nodules. In sample F20M64-DMS sample the percentage of Cu is 0.02% which results in maximum nodule count of 37.

4.1.4 Fractography:

The fractography studies in SEM revealed the mode of fracture in the DMS samples. In all the samples it was found that the mode of fracture in dual matrix structured ductile iron was mixed fracture mode. The ductile fracture and the brittle fracture was seen in all the samples. The area near to graphite nodules are seen to be all most ferrite hence the dimples were seen.

(a) (b)





(c)

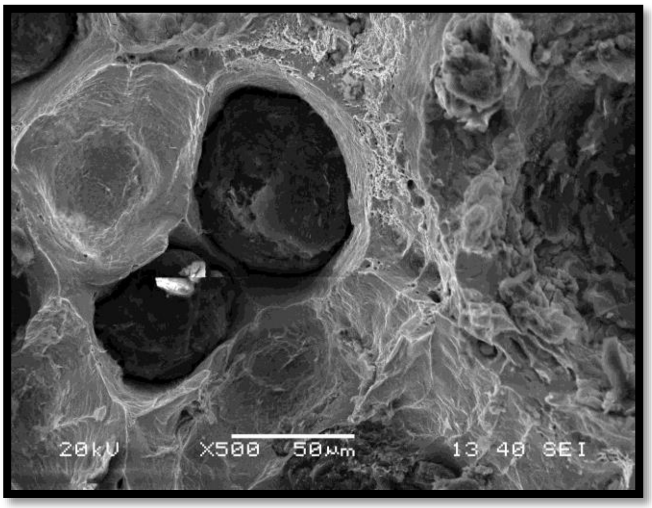
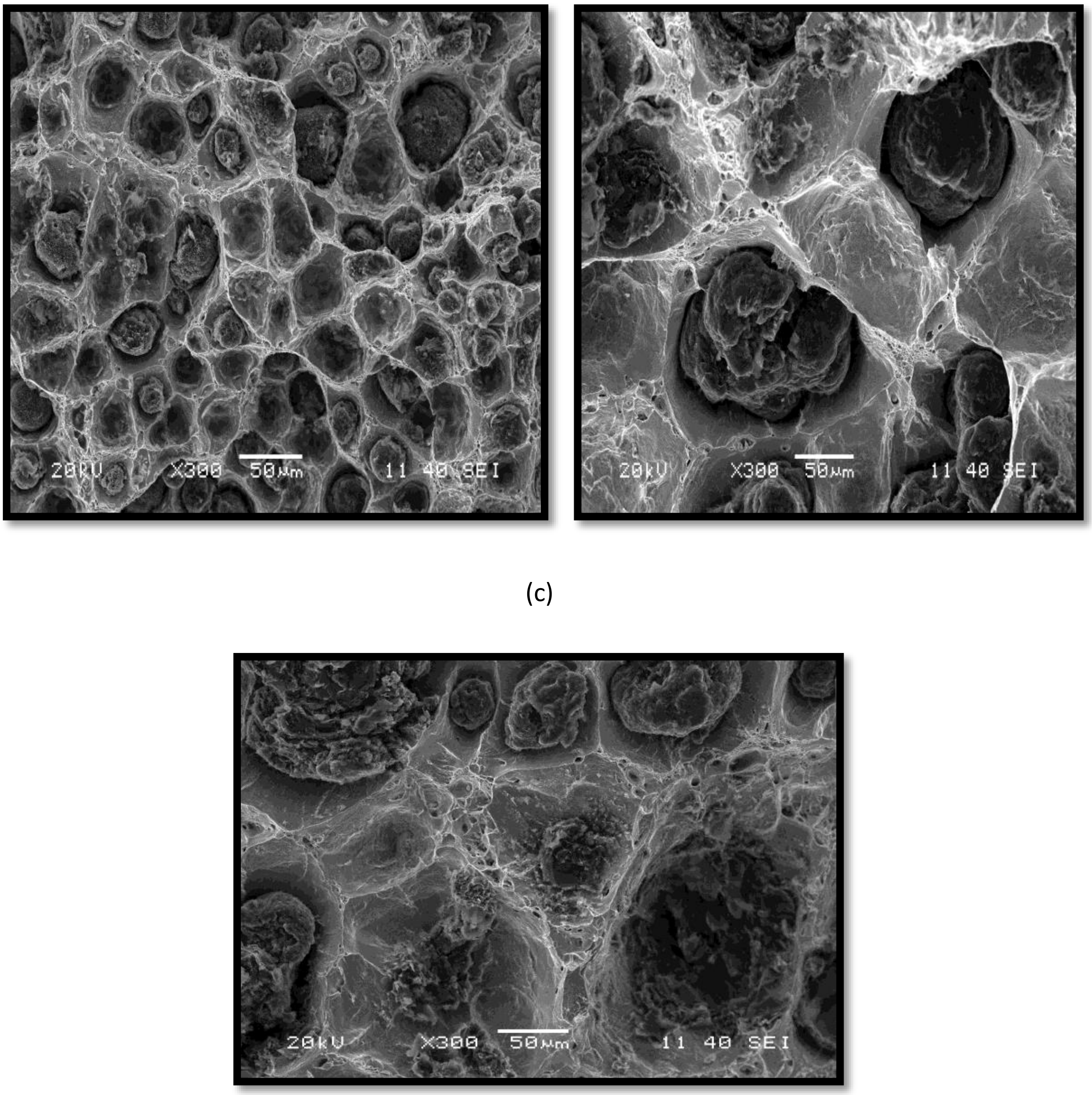


Figure-4.5 Fracture Surface of DMS ductile iron samples after tensile testing.

(a) F23M70-DMS (b) F20M64-DMS (c) F25M60-DMS

Some river markings were seen which indicates the brittle mode of fracture. In sample F23M70-DMS the martensite volume fraction is highest and hence the brittle fracture modes are higher than the other samples. In sample F25M60-DMS the dimples are more which indicates more percentage ductile fracture than other samples. From SEM analysis it is found that the tensile as well as the impact tested samples have under gone same mixed fracture mode. The fracture surface analysis was conducted at different area to reveal the both type of fracture mode.

(a) (b)



(c)

Figure-4.6 Fracture Surface of DMS ductile iron samples after impact testing.

(a) F23M70-DMS (b) F20M64-DMS (c) F25M60-DMS

Chapter-05

Conclusions

Conclusion:

This thesis reports the results of a systematic study of influence of chemical composition on ferrite and martnesite volume fraction hence mechanical properties and the microstructural observation and also fractographic studies of DMS ductile iron with different composition. The conclusions drawn from the present investigation are as follows:

1. For the DMS DI samples martensite volume fraction increases and ferrite volume fraction decrease with increase in silicon content.
2. Ultimate tensile strength, yield strength increases and ductility decreases with the increase in martensite volume fraction and decrease in ferrite volume fraction.
3. The different fracture mechanism corresponds to the different level of martensite volume fraction and observed mechanical properties. Fractrographic examination shows that DMS DI samples show mixed fracture mode.
4. The ductility increases with increase in ferrite volume fraction which resulted from increase in silicon and other ferrite stabilizer.
5. Nodule count increases with increase in percentage copper which results in increase of ultimate tensile stress and 0.2% proof stress.

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